

AMENDMENT  
U.S. Appln. No. 10/072,869

**REMARKS**

Upon entry of the claim amendments, Claims 1-15, 17, and 19-20 will be all the claims pending in the application.

Applicants have amended pages 13 and 15 of the specification consistent with page 5, Figure 5, of the Technical Bulletin of the Shell Chemical Company and consistent with Chemical Abstracts Accession No. 2001:663127 of the Polymer Preprints article by Sasaki and registry No. 178233-72-2, as noted by the Examiner at page 5 of the present Action.

Applicants have incorporated the subject matter of Claims 16 and 18 into Claim 1. Applicants have also incorporated the subject matter of Claim 21 into Claim 20. Claims 16, 18 and 21 have been canceled.

No new matter has been added.

Applicants affirm their provisional election made June 6, 2003.

Referring to the §112, first paragraph, rejection of Claims 1-5, 9-10, 12 and 14-21, Applicants respectfully request its withdrawal. In particular, the specification has been amended as requested by the Examiner at page 5 of the Action.

At the top of page 6 of the Action, Claims 1-5, 9-10, 12, and 14-21 have been rejected under 35 U.S.C. § 102(a) as allegedly being anticipated by the ACS Polymer Preprints article by Sasaki entitled "Photo-curable pressure sensitive adhesives using alkyl oxetane."

Applicants respectfully traverse and request the withdrawal of this §102(a) rejection.

A rejection based on §102(a) can be overcome by perfecting a claim to priority under 35 U.S.C. § 119(a)-(d), thereby antedating the reference. 37 C.F.R. § 1.55(a). Accordingly, Applicants are filing herewith an English-language translation of Japan Application No. 2001-34812 ("the '812 Application), from which the present application claims foreign priority. The statement that the translation is accurate will be submitted promptly hereafter. If the Examiner has not received the statement by the time he takes the present case up for action, he is kindly requested to contact the undersigned at the local telephone number listed below.

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The '812 Application was filed in Japan on February 13, 2001. Furthermore, the '812 Application fully supports the subject matter of the pending claims. Applicants refer, for example, to the claims of the '812 Application and the disclosure at Section [0028].

The Preprints article, on the other hand, was published on August 26, 2001. For the Examiner's convenience, a copy of the Preprints article is submitted herewith.

At the bottom of page 6 through the middle of page 7 of the Action, the following rejections are set forth:

Claims 1, 5, 9, and 14-21 are rejected under 35 U.S.C. § 102(b) as allegedly being anticipated by PCT Publication No. WO 00/63272 ("WO '272"); and

Claim 12 is rejected under 35 U.S.C. § 103(a) as allegedly being unpatentable over WO '272.

Applicants respectfully traverse and request the withdrawal of these rejections.

The present invention is directed to a cationically polymerizable liquid composition, and a tacky polymer obtained by cationic polymerization of the cationically polymerizable liquid composition. Independent Claims 1 and 20. The cationically polymerizable liquid composition is a homogeneous composition.

In particular, claimed solid resin (B) is compatible with the monomer mixture (A-1+A-2+A-3) at room temperature, as recited in the independent claims. Solid resin (B) is a tack-imparting resin, *i.e.* a tackifier, and adjusts the viscoelastic characteristics (at 1 Hz and 100 Hz) of the photo-polymerized polymer. Applicants refer, for example, to the second full paragraph at page 16 of the specification.

WO '272 does not disclose (anticipate) or suggest (render obvious) the homogeneous cationically polymerizable liquid composition of the present invention.

WO '272 discloses a photo-curable resin composition for photo-fabrication of three-dimensional objects. WO '272's composition does not comprise a tackifier. Applicants refer, for

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example, to the abstract for WO '272. WO '272's composition is heterogeneous because component (D) is elastomer particles with an average particle diameter of 10 to 700 nm, which does not dissolve in the monomer mixture. WO '272's heterogeneous composition results in a hard photo-polymerized polymer of impact resistance. Pages 25-27 of WO '272. The polymer obtained from WO '272 is not a tacky polymer having excellent tack properties, such as adhesion and holding power. Applicants refer to the third paragraph at page 4 of the specification.

At the bottom of page 7 of the Action, Claims 1-5, 9-10, 12, and 14-21 are rejected under 35 U.S.C. § 103(a) as allegedly being unpatentable over European Patent Publication No. 0 848 294 ("EP '294").

Applicants respectfully traverse and request the withdrawal of this §103 rejection.

The motivation or suggestion to make the claimed composition must be found in the prior art and cannot be based on Applicants' disclosure. MPEP § 2143 and In re Vaeck, 20 USPQ2d 1438 (Fed. Cir. 1991). In the present case, the assertion that it would have been obvious to use the petroleum resin in order to promote the adhesion of the blend is not found in EP '294. EP '294 does not at all mention including petroleum resin to promote adhesion. EP '294 does not disclose the softening point of the petroleum resin. EP '294 simply lists petroleum resin at page 13 as an optional additive amongst the host of optional components running from page 11, line 15, through page 14, line 14.

The present application, on the other hand, discloses the unexpected benefits of including, as a claimed component, solid resin (B) with a softening point of at least 40°C (we refer to the disclosure beginning at page 16, line 13, of the specification).

Furthermore, an alleged *prima facie* case of obviousness may be rebutted by demonstrating the unexpectedly superior results achieved by the claimed invention.

Applicants, moreover, may compare the claimed invention with prior art that is more closely related to the invention than the prior art relied upon by the Examiner. In re Holladay, 199 USPQ 516 (CCPA 1978); Ex parte Humber, 217 USPQ 265 (Bd. App. 1961).

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In the present case, there can be no closer comparison than the comparison between Example 1 and Comparative Example 1, as set forth at pages 24-31, and especially Tables 1-3. As can be seen from Table 1, the composition of Comparative Example 1 is practically the same as the composition of Example 1, except that the composition of Comparative Example 1 does not include Regalite 1090, a hydrogenated petroleum resin having a softening point of about 90°C. Thus, the composition of Comparative Example 1 is representative of the exemplified compositions in EP '294, and the composition of Comparative Example 1 is actually closest to the claimed composition (as represented by the composition of Example 1) than any of the example compositions in EP '294. All of the other components of the composition of Comparative Example 1 are the same as the components of the composition of Example 1 and are present in the same amounts (OXR-12, EKP-207, Rhodorsil 2074). The only difference is that Comparative Example 1 does not comprise any tackifier and shows poor tack strength.

Therefore, the unexpected results demonstrated at Tables 2-3 and pages 30-31 of the specification rebut the alleged *prima facie* case of obviousness.

At page 8 of the Action, Claims 1-5, 9-10, 12, and 14-21 are rejected under 35 U.S.C. § 103(a) as allegedly being unpatentable over Japan Patent Publication Nos. 11-140279, 11-152441, 10-158581, 5-171083, 5-171084, 7-62082, and 7-53711 in view of Japan Patent Publication Nos. 9-40760 and 9-328651.

Applicants respectfully traverse and request the withdrawal of this §103 rejection.

None of the primary or secondary references cited discloses or even suggests the viscoelastic properties represented by the four relationships cited in the present independent claims.

Furthermore, as mentioned above, an alleged *prima facie* case of obviousness may be rebutted by demonstrating the unexpectedly superior results achieved by the claimed invention.

The unexpected results demonstrated in the present specification rebut any alleged *prima facie* case of obviousness. That is, tacky polymers obtained by cationic polymerization of a cationically polymerizable liquid composition according to the present invention satisfy the four

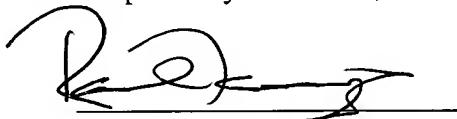
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viscoelastic characteristics relations, show no so-called "glue residue," and possess good initial adhesion, good pressure-sensitive adhesive characteristics, and high tack value. Applicants refer, for example, to page 30 and Tables 2 and 3 in the present specification.

Reconsideration and allowance of this application are now believed to be in order, and such actions are hereby solicited. If any points remain in issue which the Examiner feels may be best resolved through a personal or telephone interview, he is kindly requested to contact the undersigned at the telephone number listed below.

The USPTO is directed and authorized to charge all required fees, except for the Issue Fee and the Publication Fee, to Deposit Account No. 19-4880. Please also credit any overpayments to said Deposit Account.

Respectfully submitted,



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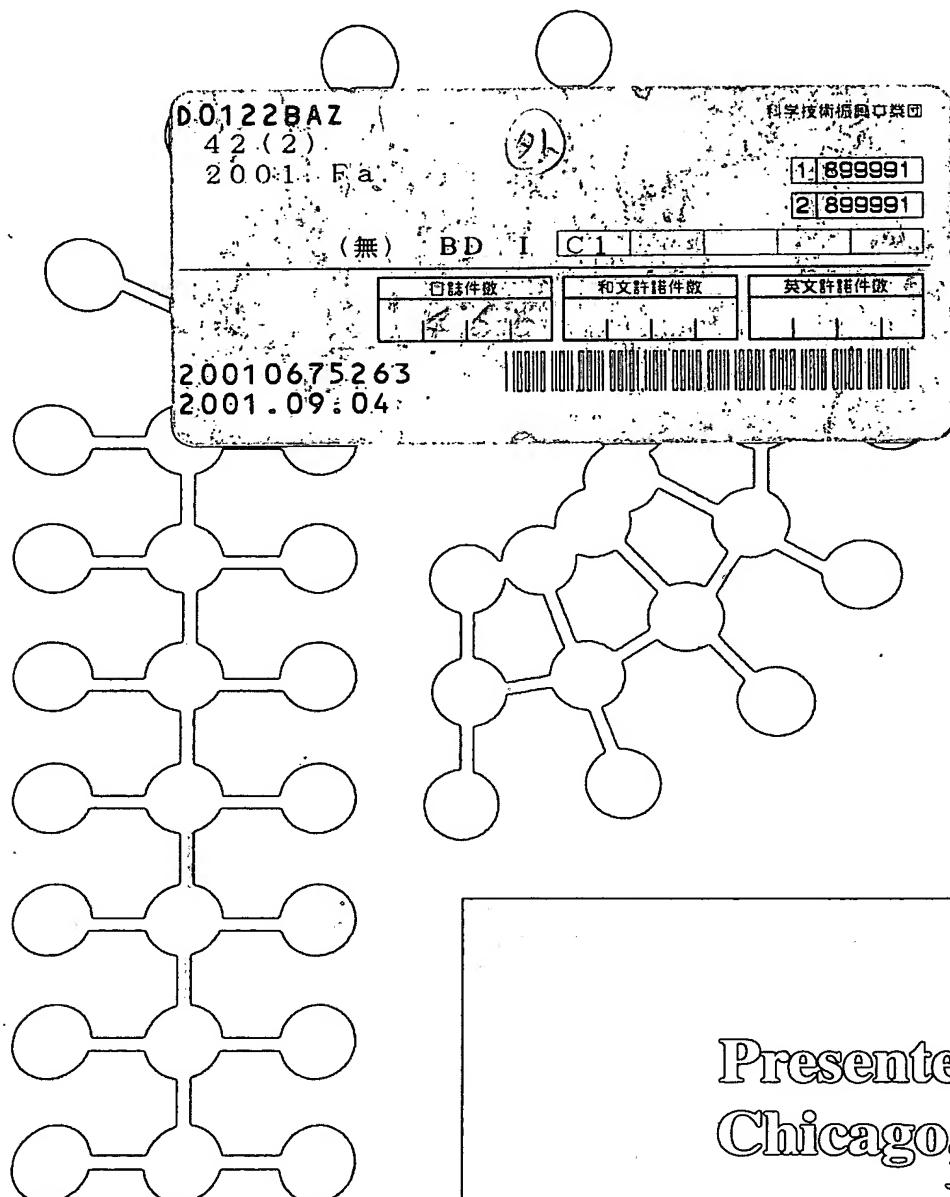
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# POLYMER PREPRINTS

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Meeting

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42 (2)

## CHICAGO, IL MEETING PROGRAM

AUGUST 26 – 30, 2001

### **MACROMOLECULAR ASSEMBLIES FOR OPTICAL AND ELECTRONIC APPLICATIONS**

*Cosponsored with Division of Polymeric Materials: Science and Engineering*  
*K. J. Wynne, J. P. Armistead, D. J. Sandman, and A. Guiseppi-Eli, Organizers*

#### *Optical Properties*

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Multifunctional organic photorefractive materials: The past and future. L. Yu

Photoinduced chirality in azobenzene containing liquid crystal polymers. P. Rochon,  
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Detection of DNA hybridization using conductive polymer layers. A. Guiseppi-Eli

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## PHOTO-CURABLE PRESSURE SENSITIVE ADHESIVES USING ALKYL OXETANE

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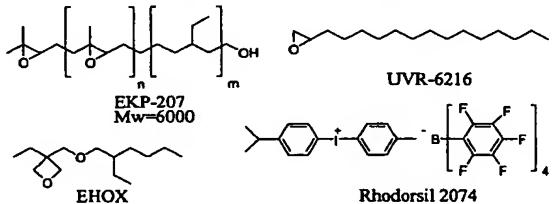
## Introduction

Pressure sensitive adhesives (PSAs) are widely used not only in industrial applications but also in our daily life, such as packaging, tapes, labels and so on, because of their excellent characteristics - easy to stick to many kind of substrates without any activation and easy to peel. Among plenty kind of PSAs, solvent- or water-borne systems are most popular. With increased environmental pressures, replacement of solvent born systems is becoming gradually more important and solvent free system is strongly desired.

To achieve a solvent free system, UV-curable type PSAs have been proposed as environmental-friendly technologies. Acrylic monomer diluted UV-curable system, which polymerizes through radical polymerization, has been mainly evaluated.<sup>1</sup> As oxygen is known to inhibit the radical polymerization, nitrogen atmosphere or lamination should be applied to complete the radical polymerization of acrylic monomers. At the same time, the residual unreacted monomers are known to cause odor problem and skin irritation.<sup>2</sup> To solve this problem of acrylic radical systems, photoinitiated cationic polymerization system has been introduced. For example, cationically curable PSA formulations consist of rather low molecular weight heterottelechelic-polymer and tackifier was introduced.<sup>3</sup> The UV-cured PSA sheet of this formulation was reported to possess good adhesive properties, still some problems seem to remain. The viscosity of the formulation is, however, rather high and more than 80°C of heat should be applied for the coating to substrates. The formulation range is also narrow due to the low compatibility of the polymers used. These problems could be a barrier to the industrial application of this system.

Oxetanes have been proved to possess many good properties, such as safety (AMES Test negative) and high reactivity.<sup>4</sup> Additional to these properties, alkyl oxetanes, having alkyl side chain, exhibited high compatibility with other monomers and very low viscosity. Using alkyl oxetanes, low viscous and high reactive UV-curable PSA formulation seems to be possible.

In this paper, the UV-curable PSA formulation using alkyl oxetane in combination with monomers listed below and adhesive properties of cured PSA sheet correlated with their viscoelastic characters were investigated.



## Experimental

**Materials.** All materials listed below were used as received.  
**EHOX:** 3-Ethyl-3-(2-ethylhexyloxymethyl)oxetane from Toagosei Co. Ltd.  
**UVR-6216:** 1,2-Epoxy-hexadecane from Union Carbide Co.  
**EKP-207:** Heterotelechelic linear polymer from KRATON Polymers.  
**Rhodorsil 2074:** Iodonium salt cationic photoinitiator from Rhodia Co.  
**P-90:** Hydrogenated petroleum resin from Arakawa chemical industries, Ltd.

**Sample Formulations.** The PSA formulation was prepared by mixing monomers, photoinitiator, and tackifier in the desired composition at 40°C. The viscosity of formulations was measured by Brookfield viscometer at 25°C.

**Photo-DSC measurement.** Heat of polymerization during photo-cationic polymerization was measured by DSC220C (Seiko Instruments Inc.) equipped with UV-1 lighting unit (200W Hg-Xe lamp, Seiko Instruments Inc.). 2 to 3mg of samples were applied to aluminum pan and irradiated UV light.

under air atmosphere. The light intensity was adjusted to 20mW/cm<sup>2</sup> at 365nm using ND filter.

**Viscoelasticity Measurement.** A 1mm-thickness PSA formulation was cured using UV irradiator equipped with 120w/cm of high pressure Hg lamp for 1 minute. The total irradiated UV energy under this condition was 2.85J/cm<sup>2</sup>. The viscoelasticity of the cured PSA sample was measured using parallel plate method by Dynamic Spectrometer (RDS II: Rheometrics Inc.). Modulus dependency for frequency was measured with frequency range of 0.1 to 10rad/sec at 25, 15, 5 and -5°C respectively. Master curve (25°C, 0.1 to 100rad/sec.) for the modulus versus frequency was made by a horizontal shift of these curves according to the shift factor derived from WLF equation.

**Adhesion properties.** The PSA formulation was coated on 50 $\mu\text{m}$  PET film (25 $\mu\text{m}$  thickness) using an applicator. The PSA sheet was prepared by curing the coated liquid formulation using a conveyor type UV irradiator equipped with 120W/cm of high pressure Hg lamp at 10min./m. of conveyor speed. The total irradiated UV energy was 135mJ/cm<sup>2</sup>. The adhesion properties of the PSA sheet, such as a peel adhesion (180°) to stainless steel (SUS) or polyethylene (PE) at 25°C and a holding power at 40°C were measured according to JIS-Z0237. Probe tack at 25°C was measured according to ASTM D-2979. Conditions for Shear Adhesion Failure Temperature (SAFT) Test were: 25mm X 25mm contact area, 500g load, 0.4°C/min. increase.

**Table 1. PSA Formulations and Their Adhesive Properties**

R.N.	1	2	3	4	5	6	7
EHOX	80	80	80	80	60	40	
UVR-6216					20	40	80
EKP-207	20	20	20	20	20	20	20
Rhodorsil 2074	1	1	1	1	1	1	1
P-90	60	80	100	120	120	120	120
Viscosity (cps) <sup>1)</sup>	390	760	1480	2870	2400	2020	1470
180° Peel <sup>2)</sup> (g/inch)	SUS	220	620	1050	1210	680	450
	PE	40	160	370	618	360	460
Holding Power <sup>3)</sup> (hrs)	>24	>24	>24	>24	>24	>24	>24
SAFT (°C) <sup>4)</sup>	164	126	117	109	91	80	60
Probe Tack (gf) <sup>5)</sup>	579	665	490	371	320	345	60

1) Measured at 25°C

2) 25µm coating was cured with 120W/cm high pressure Hg Lamp at 10m/min. conveyor speed and measured according to JIS Z-0237

3) Measured at 40°C according to JIS Z-0237

4) 500g of load and  $0.4^{\circ}\text{C}/\text{min}$ . of temp. increase

5) Measured according to ASTM D-2979

## Results and Discussion

The properties of a PSA are known to depend primarily on the viscoelastic nature of the adhesive mass.<sup>5</sup> As the rate of deformation of PSA during bonding is considered to be low, the modulus at low frequencies should be depressed, so that the composition is soft enough to flow and wet the substrate in the short time. On the other hand, on peeling the bond, the rate is high because the thickness of the adhesive is low. The modulus of a PSA should be elevated to maintain the adhesive layer at high testing rate. In the formulation of ordinary PSA, an elastomer or rubbery polymer provides the elastic component, while a tackifier constitutes the viscous component. To function properly, a tackifier must be reasonably compatible with the base polymer. If the compatibility is low, with higher amount of resin, the modulus of PSA formulation increases across the entire frequency range of 0.1 to 100 rad/sec. For a compatible tackifier, as the concentration of tackifier increases, the modulus is depressed at low frequencies and increases at higher frequency region.

The properties of the PSA formulations using EHOX, EKP-207, P-90 (tackifier) and Rhodorsil 2074 (photo initiator) were shown in Table 1. The compatibility of each component was good for these formulations and a clear liquid was obtained.

(R.N.1 to 4) The viscosity of the formulation was increased with addition of P-90. With aide of EHOX formulation, the viscosity of the formulation containing 120phr of P-90 was still low.

The storage modulus ( $g'$ ) and loss modulus ( $g''$ ) plots against frequency (0.1 to 100 rad/sec.) for the formulations are shown in Figure 1. With higher amount of P-90, the storage modulus was depressed at low frequency and increased at high frequency, which exhibited the good compatibility of the tackifier. The loss modulus was increased across the range. Tan  $\delta$  plots for the formulations are shown in Figure 2. The value was increased with addition of P-90. These high values of Tan  $\delta$  should contribute to the stress relaxation during bonding and peering.

In the formulation of ordinary PSA, adhesion properties are known to be optimized by the quantity of tackifier.<sup>5</sup> To some extent, peel strength and tack increases as the content of tackifier is increased, and reached maximum. Optimum content of tackifier for tack is lower than that for peel strength. Peel strength was increased with addition of P-90, and probe tack reached maximum with 80phr of addition. On the other hand, the SAFT was reduced with higher amount of P-90. This drop could be explained by the lower storage modulus at low frequency.

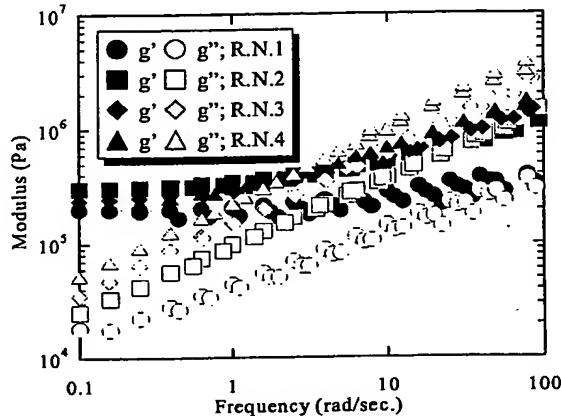


Figure 1. Storage ( $g'$ ) and loss ( $g''$ ) modulus plot for R.N.1 to 4

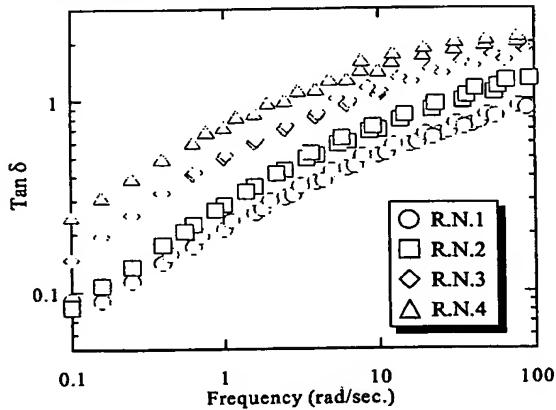


Figure 2. Tan  $\delta$  plot for R.N. 1 to 4

(R.N.4 to 7) With addition of UVR-6216, the viscosity of the formulations was reduced. Complete substitution of EHOX with UVR-6216 resulted in poor photo-polymerization (R.N.7).

The heat of polymerization of formulations was measured by Photo-DSC and the results were shown in Figure 3. In the photo-cationic polymerization of R.N.4, higher amount of exo-therm was observed in the early stage of irradiation and decreased smoothly after the peak. This smooth decrease could be explained by the fast propagation rate because of the high basicity of oxetane ring. With addition of UVR-6216 to EHOX, the peak height was reduced (R.N.5 and 6). Small amount of heat was seen for R.N.7 and the heat generation lasted for long period of irradiation. This long heat flow could be explained by the slow polymerization of oxirane ring.

As the content of UVR-6216 is increased, both of the storage modulus ( $g'$ ) and loss modulus ( $g''$ ) was depressed across the entire frequency range (Figure 4). This decrease in modulus could be explained by the remaining monomer, which should work as a softener, due to low reactivity of UVR-6216. All the adhesive properties were also depressed with high content of UVR-6216.

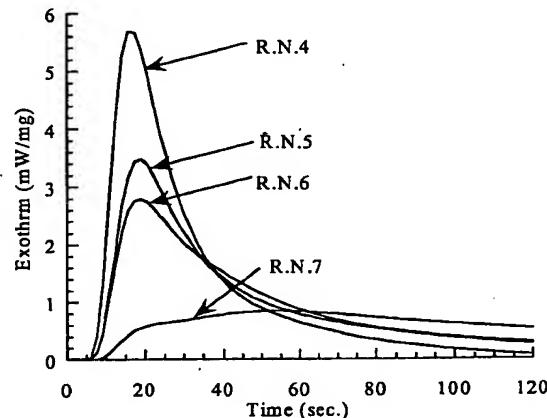


Figure 3. Photo-DSC curves for R.N.4 to 7 ( $I=20\text{mW/cm}^2$  at 365nm)

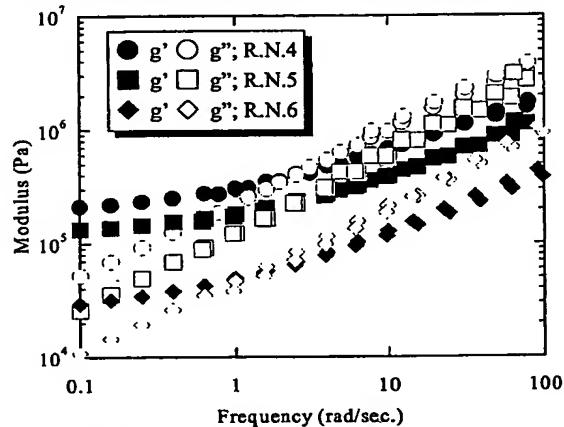


Figure 4. Storage ( $g'$ ) and loss ( $g''$ ) modulus plot for R.N.4 to 6

### Conclusions

Using mono-functional alkyl oxetane EHOX, Photo-curable PSA formulations in combination with epoxy monomers and hydrogenated petroleum resin as tackifier were investigated. The viscoelasticity and adhesion properties of the cured PSA formulations were also evaluated. The characteristics are listed below.

- 1) With aide of EHOX, uniform and clear PSA formulations with low viscosity and good reactivity were achieved.
- 2) Hydrogenated petroleum resin exhibited good compatibility with photo-polymer and was found to work as tackifier in this system.
- 3) The cured PSA sheets of the formulation were proved to possess good adhesive properties.

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